

REMARKS

I. Status of the Claims

Claims 1-28 remain pending. Claims 1, 5, 12, and 25 have been amended to correct typographical errors. It is respectfully submitted that no new matter had been introduced by virtue of this amendment.

II. Rejection under 35 U.S.C. § 102(a)

In the Office Action, the Examiner rejected claims 1-28 under 35 U.S.C. § 102(a) over U.S. Publication No. 2002/0084044 to Jollez *et al* (hereafter "the Jollez publication").

Initially, Applicants respectfully submit that present Application No. 10/010,907 was filed on November 2, 2001 and that cited U.S. Publication No. 2002/0084044, filed on November 2, 2001, was published on July 4, 2002. As U.S. Publication No. 2002/0084044 was not published before the filing date of the present application, it is not available as prior art under § 102(a).

Moreover, the specification filed on November 2, 2001 (U.S. Serial No. 10/008,454) was not filed before the filing date of the present application. As such, U.S. Publication No. 2002/0084044 is available as prior art under § 102(e) based on the November 2, 2000 filing date of the provisional application to which it claims priority (Provisional Application No. 60/245,148-copy enclosed), but not based on the specification filed on November 2, 2001. As such, only that subject matter which was disclosed in Provisional Application No. 60/245,148 may be relied upon under § 102(e). See MPEP eighth edition, Revision 2, § 2136.02, ("the disclosure relied on in the rejection must be present in the issued patent or application publication. It is the earliest effective U.S. filing date . . . of the U.S. patent or application publication being relied on as the critical reference date and subject matter not included in the

patent or application publication itself can only be used when that subject becomes public . . .”). As U.S. Publication No. 2002/0084044 claims priority to the ‘148 provisional application, which does not describe the claimed invention, U.S. Publication No. 2002/0084044 does not anticipate the present claims for the reasons set forth below.

The Examiner’s attention is respectfully directed to claim 1, which recites, in pertinent part, “[a] process for preparing a **commercially acceptable pharmaceutical grade microcrystalline cellulose** comprising . . . e) cooking the pulp in the reactor **until the pulp obtains a desired degree of polymerization, said cooking being performed at a temperature, a time, and a pressure which is a function of the desired degree of polymerization and the composition of the pulp**, the cooked pulp being hydrolyzed cellulose . . . j) **deaggregating** the hydrolyzed cellulose of step i); and k) **drying** the hydrolyzed cellulose to form microcrystalline cellulose . . .” (emphasis added).

In contrast, the ‘148 provisional application does not teach the steps of cooking the pulp in the reactor until the pulp obtains a desired degree of polymerization, said cooking being performed at a temperature, a time, and a pressure which is a function of the desired degree of polymerization and the composition of the pulp, deaggregating the hydrolyzed cellulose and thereafter drying the hydrolyzed cellulose. Moreover, the ‘148 provisional application does not teach the preparation of a commercially acceptable pharmaceutical grade cellulose as claimed. Therefore, the ‘148 provisional application does not teach each and every limitation of claim 1 and Applicants therefore respectfully request that the rejection be withdrawn. As claims 2-11, 24 and 27 depend from and incorporate the limitations of claim 1, they too are not anticipated and the rejection should be withdrawn.

Moreover, claim 5 of the present application recites “adding water to the hydrolyzed cellulose of step i) to form a solution, [and] neutralizing the solution to a pH of 5.5 or greater”,

prior to the deaggregating step. The '148 provisional application does not teach these elements of claim 5. In fact, the '148 application makes no mention of pH after filtration, let alone in the claimed range, mentioning pH only with respect to the pH during steam cooking. (See '148 provisional application at page 5, lines 4-5). Thus the rejection should be withdrawn.

Claim 12 of the present application recites, in pertinent part, “[a] process for preparing microcrystalline cellulose comprising . . . cooking the pulp in the reactor until the pulp obtains a desired degree of polymerization, said cooking being performed at a temperature, a time, and a pressure which is a function of the desired degree of polymerization and the composition of the pulp . . . j) feeding the hydrolyzed cellulose into a colloid mill; and k) drying the hydrolyzed cellulose to form microcrystalline cellulose . . .” (emphasis added).

Again, the '148 provisional application does not teach cooking pulp in a reactor until the pulp obtains a desired degree of polymerization, said cooking being performed at a temperature, a time, and a pressure which is a function of the desired degree of polymerization and the composition of the pulp. The '148 provisional application further fails to teach feeding the hydrolyzed cellulose into a colloid mill followed by drying the hydrolyzed cellulose to form microcrystalline cellulose. Therefore, the '148 provisional application does not teach each and every limitation of claim 12. Applicants therefore respectfully request that the rejection be withdrawn. As claims 13-23, 26 and 28 depend from and incorporate the limitations of claim 12, they too are not anticipated by the '148 provisional application and the rejection should be withdrawn.

III. Conclusion

A favorable action is earnestly solicited. Applicants respectfully request that a timely Notice of Allowance be issued in this application.

The Examiner is requested to contact the undersigned in the event that a telephonic interview would advance the prosecution of this application.

Respectfully submitted,
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A NEW METHOD TO PRODUCE MICROCRISTALLINE CELLULOSE HAVING A LOW DP AND ALLOWING TO KEEP THE NATURAL TEXTURE OF THE FIBERS.

FIELD OF THE INVENTION

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The present invention relates to a process of preparation of microcristalline cellulose.

DESCRIPTION OF PRIOR ART

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Canadian application No. CA 2,313,261 (JOLLEZ) describes a process for preparation of microcristalline cellulose. This process is characterised in that the pulp obtained at the end of a stage of thermo mechanical pulping is submitted to a sudden and violent depressurisation and a shear force. This process has for effect to produce a non selective fragmentation of the microcristalline cellulose resulting in the production of impurities by the oxidation during and after the explosion of the pulp.

Canadian patent No. CA 1,198,703 (DELONG) has for object a process which generates a mixture of sugar and cellulose more or less degraded. This process uses wood as a starting material and sulphuric acid, sulphurous (SO₂) or hydrochloric acid.

Canadian patent No. CA 2,137,890 (AKZO) shows the conversion of cellulose fibers, which is derived from a conventional process, into microcristalline cellulose by using benign reactivities like O₂ and CO₂. More particularly, it shows that a low degree of polymerisation can be obtained by the application of high-pressure at 140° to 180°C for 15 minutes to 5 hours on aqueous suspensions of cellulose (1/8 to 1/20) in the presence of O₂ and CO₂ in autoclaves in non-continuous mode.

SUMMARY OF THE INVENTION

5 A first object of the present invention is a process to manufacture microcrystalline cellulose having a fibrous appearance and the integrity of which is kept.

A second object of the present invention is the production of microcrystalline cellulose in the absence of any mineral acids, sulphur dioxide or carbon dioxide.

10 A third object of the present invention is the production of microcrystalline cellulose in the absence of violent non-selective depressurisation. The present process allows the application of a controlled depressurisation, which in turn permits a high yield of microcrystalline cellulose all the while limiting the production of non desirable derivatives.

15 More precisely, the process according to the present invention comprises the following steps:

- 20 a) preparation of a pulp by repulping,
b) pressing of the pulp obtained in a),
c) decompaction of the pulp obtained in b),
d) feeding of the pulp obtained in c) into a pre-heated reactor,
e) cooking of the pulp at a temperature, a time and a pressure allowing to obtain a pulp having a desired degree of polymerisation,
25 f) cooling and partial depressurisation of the reactor by purging the reactor, followed by a water injection into the jacket and directly into the reactor. This depressurisation prevents a disorganised destruction of the cells and allows to obtain a higher yield of microcrystalline cellulose, and
g) filtration of the pulp obtained in f).

30 One of the advantages provided by such a process, is that there is no disorganised destruction of the cells, like what is seen during a violent

depressurisation in the processes using a thermo-mechanical pulping step. In fact, contrary to the cases of thermo-mechanical pulping, in this new process, there is no exposition of the burst material to air, light or hot metallic sides. Thus there is no apparition, or very limited apparition of oxycellulose or non-desired
5 fonctionalisations since their formation is favoured by the presence of metals at these temperatures.

Another advantage provided by the process of the invention is that the filtration of the treated product is much faster, thanks to the absence of fragments resulting
10 from the random and non-selective breaking of the cellulose chains during the violent depressurisation used during the thermo-mechanical treatments like steam explosion treatment.

The yield at this stage is higher than 95 % with Temalfa 93.

The process of the invention also has for advantage to allow more efficient brightening or bleaching, facilitated by the absence of fines coming from the random breaking of the cells in a conventional steam treatment which retain the impurities and consume much more bleaching reactives. In such conditions, the
15
20 yield of this operation is superior to 99% and the peroxide brightens the pulp without delignifying or contributing to the purification of the surrounding impure environment, like in the case of explosive treatments. The degree of brightness of a bleached final product is much higher than in any other treatment by thermo-mechanical pulping.

25 These higher yields explain the decrease of the charge of solid and dissolved pollutants by more than half compared to a thermo-mechanical pulping process. The latter being avoided by the absence of non-selective fragmentation in the new process and by the decrease or absence of products of decomposition, which are
30 generated by the oxidation during and after the explosion in a thermo-mechanical pulping process.

DETAILED DESCRIPTION OF THE INVENTION

5 As mentioned hereinabove, the process of the invention comprises the steps of:

- a) preparation of a pulp by repulping,
- b) pressing of the pulp obtained in a),
- c) decompaction of the pulp obtained in b),
- 10 d) feeding of the pulp obtained in c) into a pre-heated reactor,
- e) cooking of the pulp at a temperature, a time and a pressure allowing to obtain a pulp having a desired degree of polymerisation,
- f) cooling and partial depressurisation of the reactor by purging the reactor, followed by a water injection into the jacket and directly into the reactor. This
- 15 depressurisation prevents a disorganised destruction of the cells and allows to obtain a higher yield of microcrystalline cellulose, and
- g) filtration of the pulp obtained in f).

During the cooking process at high temperature, the lignocellulosic material
 20 undergoes hydrolysis. The hydrolysis can be accelerated or slowed down by the presence of acids or bases during the cooking. At the same time, an oxidation can take place if the environment is favourable.

Under the effect of the temperature and the acidity of the reaction medium, an
 25 hydrolysis of hemicelluloses and lignin, if there are any left, can take place as well as for the amorphous zones of the cellulose. This hydrolysis will be more or less severe depending on the raw material, on the environment in which it is found and obviously depending on the conditions of pressure, time and temperature applied during the treatment.

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The hydrolysis can take place thanks to the organic acids such as acetic acid, which is freed during the thermal cutting of the acetyl groups of the hemicelluloses

chains. Such organic acids may serve as a catalyst for the hydrolysis of the other products, notably cellulose.

5 This phenomenon is illustrated by the fact that the pH during steam cooking, goes from about 4.5 to 3.5 rapidly enough depending on the quality of the pulp. A kraft pulp from softwood, for an equal treatment, will give a lower pH than a sulfite pulp from softwood, because of the higher content in hemicelluloses in the kraft pulp and, obviously, that depends on the severity of the applied treatment.

10 We are not talking here about the same effect, which can be provoked but which is very hard to control by the addition of known quantities of mineral acids to the reactional environment.

15 The advantages of low acidity resides on the fact that this will not cause a massive depolymerization of the cellulose like in the case of the DELONG patent who works with wood and ends up with cellulose that has been cut in a non-selective fashion therefore, giving a mix of sugars and fragments of cellulose chains in the presence of numerous degradation products like furfural and other degraded products coming from hemicelluloses or lignin.

20 An oxidation of the product present in the process can take place with more or less intensity depending on the degree of exposition, the temperature, the environment and the accessibility to the treated product. This oxidation will lead to degradation of products hence, to a product of lower quality than the desired product as well as
25 lower yields.

The non-controlled oxidation can also give birth to coloured products. It may also degrade or alter the product resulting in the production of oxycelluloses for example.

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TYPES OF CELLULOSES THAT CAN BE TREATED BY THE PROCESS OF
THE PRESENT INVENTION.

5 The cellulose employed in the process of the present invention may derive from a wide variety of cellulosic feedstock including but limited to, wood and wood products, such as wood pulp fibres, non-woody paper-making fibres from cotton, from straws and grasses, such as rice and esparto, from canes and reeds such as bagasse, from bamboos, from stalks with bast fibres, such as jute, flax, kenaf, cannabis, linen and ramie, and from leaf fibres such as abaca and sisal.

10 Suitable wood sources include softwood sources such as pines, spruces and firs, and hardwood sources such as oaks, eucalyptuses, poplars, beeches and aspens.

15 The cellulose obtained from the above cellulosic feedstock may be bleached; partially bleached or non bleached and/or may also result from chemical processes such as kraft, sulfite or alternative processes such as steam explosion treatment.

20 The present invention is concerned more particularly with the following two types of celluloses and the examples thereafter.

25 The Temalfa 93 cellulose from Tembec Company is obtained by the sulfite process from resinous trees. Given its quality, its standards of whiteness, its purity and its low content in resin, this pulp can be easily used in the production of carboxy-methyl cellulose, of methyl cellulose and of microcrystalline cellulose for the grades 100 or 200. This pulp is characterised in that it gives a degree of polymerisation of the MCC in the whereabouts of 225.

30 Temalfa 93 is the most commonly used around the world for the fabrication of microcrystalline cellulose in classical processes with acid.

The composition of the cellulose is the following:

| | | |
|---|-----------------|-------|
| | Pentosans: | 2.40% |
| | Ashes: | 0.05% |
| | S10 at 25 C: | 8.6% |
| | S8 at 25 C: | 5.6% |
| 5 | Alpha cellulose | 92,5% |

The kraft cellulose from Donohue at 100% resinous has the following composition:

| | | |
|----|------------------|-------|
| | Pentosans: | 7.00% |
| 10 | Ashes: | 0.36% |
| | Alpha cellulose: | 89%. |

TYPES OF ADDITIVES THAT CAN BE USED WITH THE PRESENT PROCESS

15 1. Antioxidants selected from the group consisting of:

- Propyl gallate,
- Hydroquinone,
- Sodium sulfite, and
- 20 - Citric acid,

or any other product having antioxidant function and that is acceptable with the desired applications of the finish products and compatible with the operation conditions.

25

2. Commercial products such as EDTA, Dequest from Monsanto, etc...

STEPS OF THE PROCESS

30 1. Repulping the cellulose in water in the presence or absence of an additive, antioxidant or sequestrant, in a reactor mixed with the recirculation pump working at a 2% to 3% consistency

2. The repulped pulp is pumped towards a pressing system such as a screw press or any other device allowing to drain and to lower the moisture of the fibre to 70% or less in weight (wet basis).
- 5 3. The humid pulp is then decompacted and aerated on a shredder or a coarse grinder.
- 10 4. The reactor is then pre-heated to the temperature desired or to any other temperature chosen to reduce the condensation due to the heating of the walls during the treatment. This is done via the jacket or by injecting vapour directly and then emptying it before opening it to charge it.
- 15 5. The cooking reactor is then fed with wet grounded pulp and in a way to obtain a filling rate of approximately 30%. For an apparatus working in continuous mode, the feeding is done through an airlock or by any other mechanism allowing to feed a vessel that is under pressure for example a co-axial system.
- 20 6. The reactor is then closed when the apparatus in question works in batch. Vacuum can be applied before the steam feed to purge the gases present, such as air.
- 25 7. The reactor is then fed with steam directly up to a predetermined pressure. This method allows to rapidly reach a temperature between 200 and 235°C.
8. A purge of non condensables, through the top of the reactor, in the case of a batch reactor is desirable if the purge in step 6 was not carried out. Furthermore, steam must be re-introduced in the reactor to maintain the work in pressure.
- 30 9. The cooking is maintained during 4 to 25 minutes depending on the nature of the cellulose and the chosen working temperature. The goal is to reach a stable

degree of polymerisation indicative of reaching the cellulose having the DP desired for MCC.

10. The reactor is then rapidly cooled by an injection of water in the jacket and in the reactor itself, in the case of work in batch mode. A preliminary depressurisation of the excess vapour can also be carried out before the injection of cooling water.

11. In the case of continuous reactor, the treated product is pushed to one or several partially decompressed chambers for partial decompression. This insures the transport of the product towards the exit, without causing any explosion. The product is thereafter cooled down by water injection and further transported for the next step.

A variant of the decompression chambers may be carried out by means of a set of screw spindles and/or gears and/or inverted pump. This variant insures a rapid cooling of the product by a partial decompression with no explosion of the latter.

12. The mixing can then start and the reactor is emptied around 60°C by adding water to recuperate all the cellulose present in the reactor.

13. When the treated pulp is a pulp of sulfite or bleached kraft quality, it is sent directly to filtration before going to "brightening" and/or bleaching.

14. In the case of a pulp of intermediate quality, it goes on a caustic soda solution that is diluted in a way to eliminate leftover lignin and other impurities present, it is filtered, then washed before being sent to bleaching, which will be done according to the initial quality of the starting cellulose.

15. After filtration, the product is brightened with hydrogen peroxide in the following conditions:

Peroxide: 2% w/w on dry mass;

Magnesium sulfate: 0.5% w/w on dry mass; and
Sodium hydroxide: 0.5% w/w on dry mass.

The treatment could be done between 60 and 120°C and done under air or oxygen
5 pressure reaching up to 120 psi.

16. The brightening and bleaching process can be adapted in function of the
quality of the initial product, and in the more extreme cases, known bleaching
methods can be used, such as hypochlorite or chlorine dioxide bleaching.

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17. The bleaching consistency will preferably be 25% but this can also be done at
lower consistencies.

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18. The bleached pulp is filtered and may be used as such or in a dry start for new
applications comprising a new generation of microcrystalline cellulose of fibrous
appearance, but having the same specifications as a classical microcrystalline
cellulose in crystallinity index, DP.

20

19. The filtered bleached pulp can also be homogenized in water at a consistency
going from 0.5 to preferably 3% and then filtered and washed to rid the residue of
bleaching reactives. This operation is done with an apparatus of the "blender"
type, which allows the separation of microcrystalline cellulose particles to give non
colloidal microcrystalline cellulose.

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20. After filtration, the suspension obtained is brought to a dryer of the type "spray
dryer" to obtain the size required in the desired dryness of classical
microcrystalline cellulose.

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RESULTS IN YIELD PRODUCTION OF MCC OBTAINED BY THE PROCESS OF
THE INVENTION

| | | |
|--|----------|-------|
| | ALPHA 93 | KRAFT |
|--|----------|-------|

| | | |
|-------------------------------------------|------|------|
| Repulping | 100 | 100 |
| Hydrolysis and washing | 95,0 | 88,0 |
| | | |
| H ₂ O ₂ and washing | 99,0 | 99,0 |
| | | |
| NaOCl and washing | | 99,0 |
| | | |
| Homogenization | 99,5 | 98,5 |
| | | |
| Drying | 99,5 | 99,0 |
| Total Yield | 93,1 | 84,1 |

There is an increase in the yield of the alfa-pulp of 20% and an increase in the yield of the kraft pulp of 23% compared to the process by thermo-mechanical pulping using steam explosion treatment.

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EXAMPLES OF TRIALS CARRIED OUT BY THE PROCESS OF THE INVENTION

- A) TEMALFA 93 cellulose on a small scale without additives
- 10 B) TEMALFA 93 cellulose on a small scale with additives
- C) Kraft cellulose on a small scale
- D) TEMALFA 93 on a commercial scale.

A - EXAMPLE 1: TEMALFA 93 cellulose

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1kg of Temalfa 93 cellulose was repulped at a consistency of 2.5% in water, then partially dried with the help of a press and coarsely ground to obtain a residual moisture of 60.3%.

From the above obtained product, 229 g (equivalent to 90.913 g of cellulose) were introduced in a 24 litres reactor pre-heated with saturated steam. The steam is then introduced directly from the bottom of the reactor and a rapid purge is carried out to rid the non condensables.

5

Within 1 minute the product is submitted at a temperature of 220°C and is maintained for 13 minutes at this temperature. The pressure is then partially released, pressurised cold water is injected in the reactor in such a way as to allow rapid cooling of the pulp and the mixing is initiated to ensure the discharge and to carry on the treatment. The washed filtered product (252 g at 65.7% moisture) is white but slightly greyish.

10

The pH of the filtered solution is 5.3.

15

In a sample of 59,7 g a brightening with hydrogen peroxide was carried out with 2% peroxide in the presence of 0.5% magnesium sulphate on a dry pulp at a pH of 10.5. The operation was carried out for 1 hour at 60°C.

After filtration and washing, 56.7 g of pulp is recovered (64.2% moisture).

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A homogenization of 55.7 g of brightened pulp gives, after filtration and washing, 50.7 g of pulp at 60.8% moisture (19,9 g of dry product).

ANALYSIS:

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DP (Degree of Polymerisation) = 214

Cr.I (Cristallinity Index) = 84,6

MS (Microcrystal Size) = 46,6 Å

30 B - EXAMPLE 2: Temalfa 93 cellulose with additives.

A solution of 1% sodium sulphite is used in a ratio of 20/1 on 100 g of Temalfa cellulose. After pressing and coarse grinding, 214 g of soaked cellulose at 75.3% moisture is introduced into the pre-heated reactor.

- 5 The product is treated as in the example 1 for 12 minutes. After filtration and washing, we obtain 363 g of pulp at 75.3% moisture is obtained and the pH of the filtrate is 4.3.

- 10 357 g of bleached pulp obtained above is brightened with peroxide at the same conditions as in example 1. After washing and filtration, 253.3 g of pulp is recovered (moisture = 65.5%).

- 15 A homogenisation is carried out with 250 g of brightened pulp described above and after filtration and washing, 237.7 g of pulp is recovered (64% moisture).

ANALYSIS:

DP = 219

Cr.I = 88,9

- 20 MS = 46,6 Å

C - EXAMPLE 3: kraft cellulose

- 25 210 g of kraft cellulose humidified at 55.8% is treated at 220°C for 13 minutes.

After filtration and washing, 366.4 g of cellulose are recovered at 77.7% moisture. The pH of the filtered solution is 4. The cellulose obtained is coloured, light brown / caramel.

- 30 A brightening step is carried out with the same conditions as previously described. A bleaching step is then carried out with hypochlorite with 1% hypochlorite on dry cellulose at a pH of 11 at 40°C during 2 hours. The filtered bleached product has a

weight of 237.5 g and a humidity of 66.2%. The homogenization allowed the recovery of 240.4 g of pulp at 67.1% humidity.

ANALYSIS:

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DP = 224

Cr.I = 88,8

MS = 43,1 Å

10 D - EXAMPLE 4: Example on a Commercial Scale

120 kg of Temalfa 93 cellulose was repulped in the reactor mixed with cold water at a consistency of 3%. The operation is done in 6 steps of repulping of 20 kg each.

15 The pulp is then sent to a screw press of Atara/Spirac Spiropress U-260 brand to be dried up to a residual humidity of approximately 65%. The wet cellulose obtained goes through a moulding granulator that will decompact it.

20 The product obtained is loaded in a cylindrical stainless steel reactor. The reactor's volume is 2 cubic meters. After having closed the reactor, it is directly fed with steam to obtain the pressure required for the treatment. In just a few minutes the temperature into the reactor reached 220°C.

25 After 12 minutes of cooking at 220°C, water is injected in the reactor in order to lower the temperature rapidly and allow a discharge of the cooking product. The discharge of the reactor is done several times with water injection to allow for a complete recuperation of the product.

4 cubic meters of water are required to realise this operation.

30

A rotating filter of 0.9 meter diameter and 0.6 meter length is then used for the filtration and the washing of the cellulose that is obtained.

The product has a fibrous aspect, resulting from a non-destructive process, is whitish.

5 ANALYSIS:

DP = 214

Cr.l = 85,2

MS = 46,6 Å.

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Applications of the MCC obtained by this process

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The bleached product that went through the blender and that was spray dried, has similar applications as the classical applications for MCC PH 101, that is:

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Tableting (excipient with bonding properties)

Cream used in pharmaceuticals and cosmetics

Fat replacer (lipid free ice cream and mayonnaise)

Chromatography support

Complexation with transitional metals for enzyme immobilization

25

For the bleached product, untreated at high speed, the application hereafter gives new products related to the form and characteristics of fibrous microcrystalline cellulose. This cellulose is of very high purity and serves as a support for a new type of catalysts.

30

Since the structure of the product has a fibrous aspect and that, contrary to classical MCC, many OH groups from the anhydroglucose molecule are not available, they will not react with the metals used to obtain a catalyst. Furthermore, in mixing this preparation with inorganic products during a sufficient mixing and drying time, the distribution of the active sites in the sphere formed

then dried and charred, will be different than the one obtained with a classical microcrystalline cellulose conferring new properties to the finished product. The spherical substrate of the catalyst, after charring, contains holes of controlled dimension making it different than the one obtained with colloidal MCC or with
 5 ground cellulose, which is, on top of that, limited by its initial inferior quality.

PARTICULARITIES OF THE NEW PROCESS TO OBTAIN
 MICROCRYSTALLINE CELLULOSE DEVELOPPED BY KEMESTRIE.

- 10 - Vapour cooking of humidified cellulose that is saturated in water.
- Cooking without any mineral acids or dioxides.
- Presence or absence of additives (e.g. antioxidant).
- 15 - No explosion of the treated product.
- It is applicable to many types of cellulose of deciduous or resinous trees.
- 20 - Cooking of the humidified cellulose with saturated vapour.
- Controlled cooking allowing to obtain the desired degree of hydrolysis of the cellulose.
- 25 - Very short time of treatment thanks to the instantaneous heating of the cellulose with saturated vapour.
- Limited vapour consumption that is 1 to 1,2 ton of vapour per ton of dry cellulose.
- 30 - Contrary to the thermo-mechanical pulping, in this new process we do not have exposition of burst material to air, to light, or to the hot metallic sides with the possible apparition of oxycelluloses whose formation is favoured in the presence

of metals at these temperatures. Moreover, we know that when the substance is subjected to violent depressurisation such as going from 350 psi to atmosphere pressure in a few fractions of second, like in the case of thermo-mechanical pulping, the substance is treated in a destructive fashion. This process also has an abrasive effect on the material of the reactor located near the exit, thus increasing the chance for the burst treated product to be contaminated with metallic particles.

- The addition of certain cooking additives can help to avoid even more oxidation of the cellulose and its impurities.

- Very low formation of colour on a treated product with the recommended process.

- Increased efficiency of washing (which means the reduction of water quantities used).

- A degree of brightness of the finished bleached product higher than any other treatment by steam explosion.

- If need be, a homogenization of the finished product can be carried out and the breaking of the cellulose chains is done in a methodical manner contrary to what is done by classical thermo-mechanical pulping with the random explosion of cells as well as with the shear and the impact produced by the violent depressurisation.

- More precisely, with the alpha 93 pulp the yield of the initial dry pulp is 95% at the hydrolysis including the washing whereas with an explosive process where in the best of the cases as disclosed in patent no. CA 2313261 this yield goes down to 87% in similar conditions.

- With kraft pulp, the yield of recuperation at the same level is of 88% against 83% by steam explosion treatment.

[illegible]

CLAIMS

1. A process to obtain microcrystalline cellulose characterised in that it contains the following steps:
- 5 a) preparation of a pulp by repulping,
b) pressing of the pulp obtained in a),
c) decompaction of the pulp obtained in b),
d) feeding of the pulp obtained in c) into a pre-heated reactor,
e) cooking of the pulp at a temperature, a time and a pressure allowing to obtain a
10 pulp having a desired degree of polymerisation,
f) cooling and partial depressurisation of the reactor by purging the reactor, followed by a water injection in the jacket, and
g) filtration of the pulp obtained in f).
- 15 2. A process according to claim 1 characterised by the addition of antioxidants during the cooking.
3. A process according to claim 1 or 2 characterised by a cooking temperature varying from 210 to 235°C.
- 20 4. A process according to any one of claims 1 to 3, characterised by a repulping stage carried out at a consistency of 2 to 3%.
5. A process according to any one of claims 1 to 4, characterised by a cooking
25 time which is between 4 and 25 minutes according to the desired degree of polymerisation.
- 30 6. A process according any one of claims 1 to 5, characterised by a stage following cooking where the cellulose is received on a solution of diluted caustic soda which has for effect to eliminate the left over lignin and other impurities present in the cellulose.

7. A process according to any one of claims 1 to 6, characterised by a bleaching treatment.

8. A process according any one of claims 1 to 7, characterised by a temperature
5 during the bleaching treatment which is between 60 and 120°C where the air pressure or oxygen reaches 120 psi and that the treatment consists in a mixture of peroxide, magnesium sulphate and sodium hydroxide.

9. A process according to any one of claims 1 to 8, further comprising a
10 homogenisation step to obtain non colloidal MCC.

10. A process according to any one of claims 1 to 9, characterised by a drying
stage after washing and filtration which is done with the help of a dryer of the type
15 spray-dryer.

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500 μ m (50X)

TEMALFA 93 TEM CELLULOSE

Tem α

00E011-8h15h209

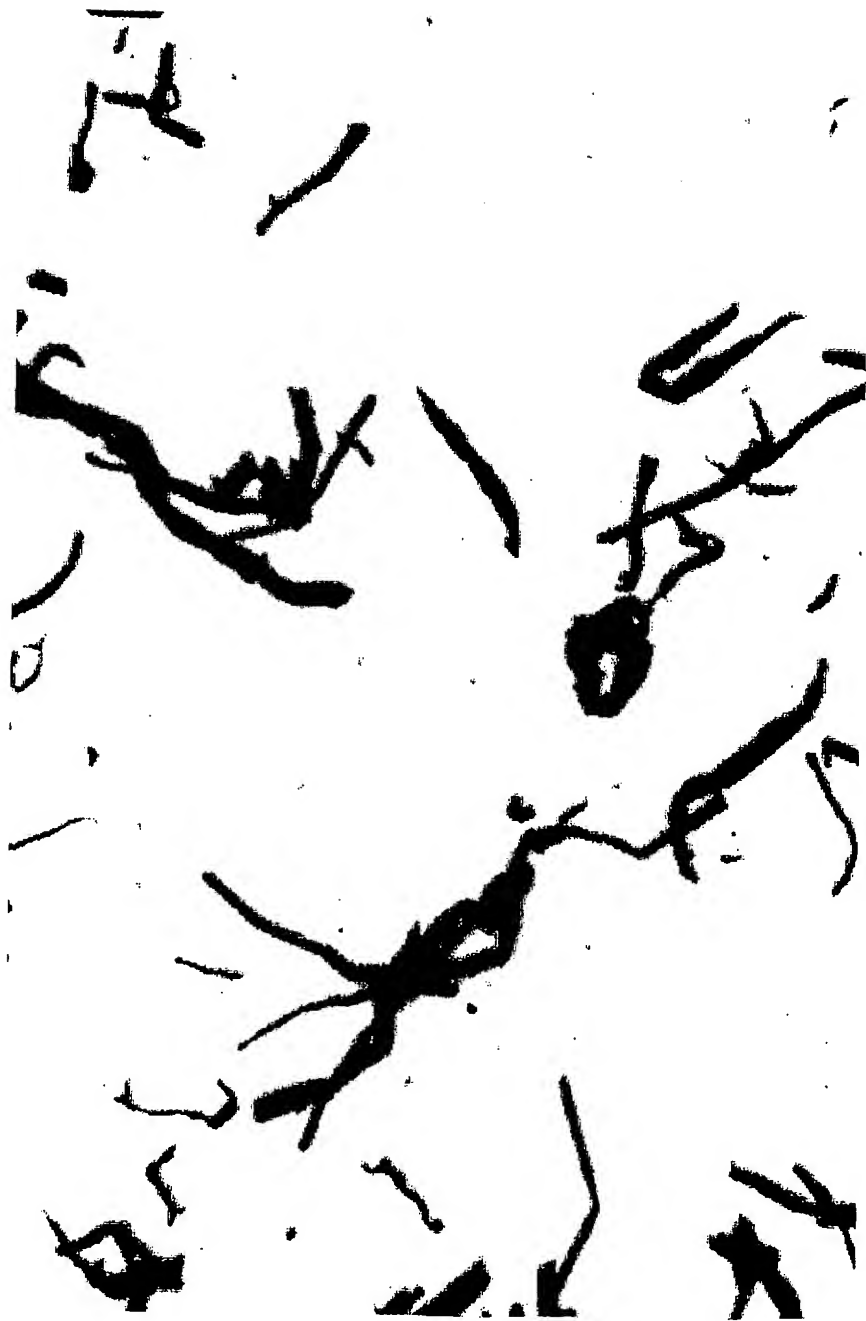


Tα VC12

→ 250 μm (100X)

TEMALPHA CELLULOSE TREATED BY A STEAM EXPLOSION PROCESS

002011-84154203



500 μm (50X)

T α 11N

TEMALPHA 93 CELLULOSE TREATED BY PROCESS OF THE PRESENT INVENTION

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